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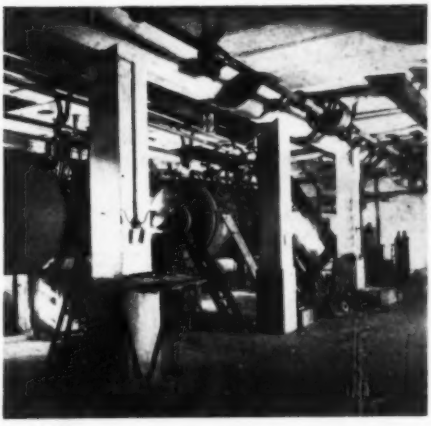

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VOLUME IV DECEMBER, 1927 NUMBER 12

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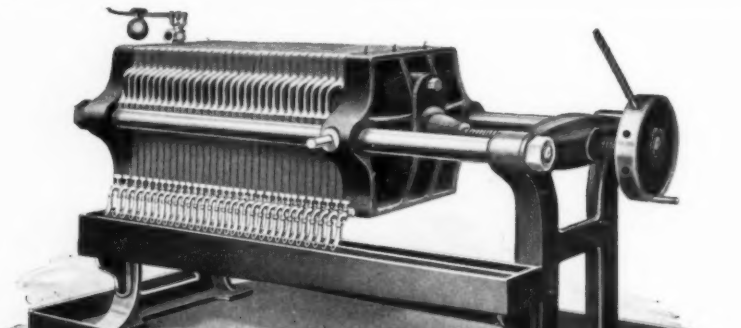

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OIL & FAT INDUSTRIES

VOLUME IV DECEMBER, 1927 NUMBER 12

The Chemical and Physical Characteristics of Cod Liver Oil

Results Obtained from Examination of Over One Hundred Samples of Known Origin

BY ARTHUR D. HOLMES* and WALTER Z. CLOUCH*

NUMEROUS investigators have shown that cod liver oil is the richest known natural source of fat-soluble vitamins. Since this fact became generally recognized, there has been a constantly increasing tendency to evaluate cod liver oil very largely on the basis of its vitamin content. Of late this tendency to grade cod liver oil solely by its vitamin content has led many to judge the value of cod liver oil largely, if not wholly, by the amount of vitamin D that it contains. In this connection, it is perhaps well to call attention to the fact that there is no official method for the determination of vitamin D and that the method for determining vitamin A appearing in the present edition of the United States Pharmacopœia is optional. As a matter of fact, at the present moment the only official standard for cod liver oil is embodied in the specifications set forth in the United States Pharmacopœia for the chemical and physical characteristics of cod liver oil. Furthermore, it may not be amiss at this point to call attention to the fact that the Federal Government insists in no uncertain terms that cod liver oil labeled as such cannot be lawfully sold for consumption by men, animals, or poultry unless it is in accord with the specifications for the chemical and physical characteristics of cod liver oil required by U.S.P.X.[1]

From the foregoing, it is evident that information concerning the chemical and physical characteristics of present day cod liver oil is of considerable interest. In some localities manufacturing conditions have changed very materially during the past years. Present day manufacturers of medicinal cod liver oil insist on strictly fresh cod livers. The livers are cooked promptly and the oil that is obtained is stored out of contact with the air. Inasmuch as this procedure is materially different from the manufacturing and storage conditions previous to the last few

*Research Laboratories, the E. L. Patch Company, Boston, Mass.

[1] The Pharmacopœia of the United States of America. Tenth Decennial Revission. J. B. Lippincott Company, Philadelphia, Pa. P. 263-264.

years, the question very materially arises as to whether the present day medicinal cod liver oil is more uniform than that of former times.

In a study of the nutritive value and market quality of cod liver oil, Drummond[2] and co-workers found that the "growth dosage" or vitamin content of the oils under consideration varied over wide limits. A later paper by one of us[3] reported the results of a vitamin study of a number of medicinal cod liver oils procured in the open market. In that study it was found that the oil having the highest vitamin content had a potency of ten times that of the oil with minimum vitamin content. It is evident from data of this character that the vitamin potency of medicinal cod liver oil is not at all uniform. In fact, it is becoming quite generally recognized that the vitamin potency of any brand or grade of cod liver oil varies from lot to lot. In view of the wide variation in the vitamin potency of cod liver oil, the question naturally arises as to whether present day cod liver oils have uniform chemical and physical characteristics. Accordingly, this study was undertaken to secure definite data concerning the chemical and physical characteristics of cod liver oil produced under commercial conditions.

Origin of Oils Studied

In order that the results of this investigation should be of general application, samples of cod liver oils were obtained from plants located all along the coast from New York to Labrador. A total of 111 samples of cod liver oil manufactured along the eastern North American coast were studied. These samples were collected during the period July 1926—July 1927, and, therefore, represent oils manufactured at all seasons of the year. Obviously, there are included in this group of samples, oils that were manufactured at all stages of the reproductive cycle of the cod fish. Accordingly, it was hoped that this study would yield general information concerning what might be termed average American cod liver oil.

Inasmuch as the samples of oil under consideration were obtained from different manufacturers, complete information is not available concerning all the details of the manufacturing process. However, it is probably safe to assume that practically all the oils under consideration were made by the direct steam process. All oils coming from Newfoundland were obviously made by the direct steam process since the Newfoundland Government has ruled that only this process may be used in that country. Furthermore, the direct steam process is now largely used in New England, New Brunswick, Nova Scotia, and along the Gaspe Peninsula. In some isolated localities, a certain amount of "pot oil" is produced by the direct fire process. It is, therefore, possible that a small amount of "pot oil" may have been included in some of the oils represented by the samples studied. In general, one may assume that the oils studied were separated from fresh cod livers by cooking at approximately boiling temperature in contact with whatever water was derived from condensing steam.

Ordinarily it is rather too diffi-

[2] The Use of Cod Liver Oils in the Feeding of Farm Animals, J. C. Drummond, S. S. Zilva, and J. Golding, Jour. Agri. Science, Vol. XIII, April, 1923, p. 153.

[3] Studies of the Vitamin Potency of Cod Liver Oils—X—Vitamin Potency of Medicinal Cod Liver Oils—Arthur D. Holmes, Ind. & Eng. Chemistry, Vol. XVI, No. 11, Nov., 1924.

cult to convey a definite conception concerning color by the use of terms which are of a general rather than a specific nature. The color of the oils in this study ranged from a very light or pale yellow to one or two samples which were of a slightly reddish yellow color. In general, however, the oils had what might be termed "a light yellow color."

In the tests which are reported below, all the oils were chilled to 0°C. Since no cloudiness was observed in the samples at this temperature, it is evident that all the oils under consideration were "cold pressed" or "non-freezing oils." Such oils are ordinarily produced by chilling crude cod liver oil to 0°C. At this temperature the crude oil becomes a semi-solid mass which is then pressed to remove the "stearin" that has solidified at freezing temperature. As a matter of fact, the so-called "stearin" of commerce is not pure cod liver stearin for when the stearin solidifies it occludes within it a certain amount of non-freezing cod liver oil. Thus commercial "cod liver stearin" is a mixture of stearin and cod liver oil.

Experimental

In the routine analysis of the cod liver oils, the methods described in detail in the U. S. Pharmacopœia[1] were followed in the determination of the specific gravity, saponification value, iodine number, free fatty acid content, and the percent of unsaponifiable matter. In addition, the refractive index of all the oils was determined by means of a Zeiss refractometer and the results obtained are included in the following table so that the data concerning the chemical and physical characteristics of the oils under consideration may be as complete as possible.

[1] The Pharmacopœia of the United States of America. Tenth Decennial Revision. J. B. Lippincott Co., Philadelphia, Pa. P. 263-264.

THE CHEMICAL AND PHYSICAL CHARACTERISTICS OF COD LIVER OIL

Sp. Gr.	Ref. Ind.	Sapon. Value	Iodine Number	F. F. A. Per cent	Unsapon. Per cent
0.922	1.4781	185.6	159.0	1.65	1.28
0.920	1.4780	189.1	165.7	1.54	1.32
0.921	1.4782	188.4	165.8	1.53	1.40
0.922	1.4780	190.8	167.4	0.75	1.21
0.923	1.4780	186.3	167.9	0.74	1.14
0.922	1.4780	187.9	156.4	0.78	1.26
0.923	1.4778	188.5	162.7	0.91	1.08
0.922	1.4778	187.6	163.2	0.74	1.11
0.923	1.4780	189.4	164.3	0.78	1.21
0.923	1.4778	188.4	163.9	0.77	1.10
0.923	1.4778	189.7	165.4	0.79	0.97
0.922	1.4780	186.7	164.4	0.87	1.00
0.921	1.4780	181.9	165.2	0.91	1.19
0.923	1.4780	188.7	157.0	0.64	1.10
0.921	1.4775	183.2	154.4	1.27	1.15
0.920	1.4775	186.2	155 7	1.02	1.02
0.920	1.4778	184.9	156.3	0.80	1.01
0.921	1.4780	184.0	151.6	0.77	1.04
0.921	1.4780	186.9	157.7	0.74	1.08
0.921	1.4780	185.3	151.4	0.86	1.08

Sp. Gr.	Ref. Ind.	Sapon. Value	Iodine Number	F. F. A. Per cent	Unsapon. Per cent
0.922	1.4780	184.4	156.3	0.65	1.03
0.922	1.4780	184.9	154.8	0.62	1.15
0.922	1.4783	183.4	160.1	0.61	1.19
0.922	1.4780	185.5	155.1	0.65	1.10
0.922	1.4782	185.8	158.0	0.58	1.14
0.922	1.4781	184.1	155.3	0.55	1.14
0.922	1.4780	186.5	157.0	0.53	1.05
0.922	1.4780	185.6	160.1	0.51	1.13
0.922	1.4780	185.4	159.4	0.67	1.13
0.922	1.4780	186.8	158.6	0.63	1.13
0.922	1.4780	185.1	159.6	0.60	1,10
0.922	1.4780	185.1	159.6	0.60	1.10
0.923	1.4786	186.7	158.5	0.58	1.12
0.922	1.4780	185.2	156.7	0.57	1.10
0.920	1.4780	185.8	158.9	0.59	1.13
0.922	1.4780	184.6	158.9	0.60	1.12
0.921	1.4782	188.7	162.3	0.54	1.09
0.921	1.4778	186.8	161.1	0.79	1.02
0.922	1.4778	186.6	158.9	0.68	1.06
0.921	1.4778	186.2	149.5	0.66	1.08
0.921	1.4778	184.9	156.3	0.63	1.12
0.921	1.4776	187.3	147.6	0.50	1.06
0.921	1.4775	187.5	155.2	0.51	1.03
0.921	1.4777	186.5	156.6	0.48	1.04
0.922	1.4775	188.3	157.2	0.49	1.08
0.921	1.4775	187.9	157.6	0.53	1.08
0.922	1.4779	188.9	164.5	0.48	1.04
0.922	1.4772	187.2	159.2	0.49	1.05
0.926	1.4820	187.8	181.3	0.41	1.00
0.927	1.4805	188.6	183.1	0.31	1.01
0.923	1.4778	187.8	159.3	0.44	1.01
0.922	1.4778	187.9	158.2	0.41	1.04
0.923	1.4785	180.8	156.1	0.58	1.21
0.923	1.4785	180.9	156.9	0.60	1.25
0.921	1.4785	188.0	154.9	0.36	1.22
0.922	1.4785	187.9	158.5	0.36	1.27
0.924	1.4790	183.9	159.7	0.30	1.16
0.923	1.4790	184.2	156.3	0.30	1.17
0.924	1.4785	186.7	154.9	0.30	1.11
0.924	1.4785	186.5	160.7	0.30	1.20
0.927	1.4800	187.8	173.6	0.29	1.19
0.926	1.4806	187.1	175.3	0.30	1.15
0.925	1.4805	187.0	174.0	0.35	1.20
0.925	1.4805	186.8	173.6	0.29	1.12
0.925	1.4805	186.4	173.2	0.28	1.15
0.925	1.4805	186.5	174.7	0.29	1.23
0.925	1.4805	186.6	172.8	0.29	1.25
0.925	1.4805	187.0	174.3	0.29	1.12
0.925	1.4805	187.0	174.1	0.27	1.16
0.925	1.4803	186.0	171.2	0.36	1.24
0.925	1.4809	186.3	171.1	0.36	1.23
0.925	1.4805	186.1	171.1	0.34	1.23
0.925	1.4805	186.2	171.3	0.29	1.23

Sp. Gr.	Ref. Ind.	Sapon. Value	Iodine Number	F. F. A. Per cent	Unsapon. Per cent
0.925	1.4807	186.4	171.2	0.29	1.17
0.925	1.4807	187.3	172.0	0.28	1.20
0.925	1.4807	186.8	172.4	0.29	1.27
0.925	1.4805	186.7	172.6	0.30	1.29
0.925	1.4800	186.6	171.5	0.29	1.19
0.925	1.4802	186.6	171.3	0.28	1.11
0.925	1.4800	186.8	170.0	0.29	1.18
0.925	1.4798	186.0	173.9	0.29	1.19
0.925	1.4798	185.7	174.2	0.30	1.12
0.925	1.4798	186.5	172.5	0.30	1.19
0.925	1.4798	186.6	173.2	0.29	1.18
0.925	1.4798	186.5	169.4	0.37	1.10
0.925	1.4793	186.0	169.7	0.36	1.08
0.925	1.4791	186.7	170.7	0.30	1.14
0.923	1.4783	183.7	172.2	0.34	1.14
0.925	1.4790	186.3	171.1	0.30	1.07
0.922	1.4794	184.1	169.0	0.47	1.17
0.922	1.4794	183.4	168.2	0.43	1.18
0.923	1.4790	184.5	169.0	0.36	1.19
0.923	1.4796	182.7	170.6	0.36	1.19
0.922	1.4789	184.3	172.3	0.45	1.19
0.921	1.4794	189.6	166.0	0.44	1.19
0.923	1.4795	184.4	168.6	0.39	1.36
0.923	1.4790	184.8	171.3	0.41	1.33
0.923	1.4790	184.2	168.1	0.41	1.31
0.922	1.4790	183.8	167.0	0.50	1.16
0.923	1.4790	183.8	165.6	0.48	1.19
0.922	1.4790	185.3	177.1	0.47	1.26
0.923	1.4781	181.1	167.6	0.46	1.27
0.922	1.4782	184.0	164.0	0.55	1.35
0.923	1.4782	183.3	165.9	0.58	1.32
0.923	1.4782	184.0	167.0	0.58	1.35
0.923	1.4787	185.1	166.2	0.91	1.32
0.923	1.4785	184.1	167.1	0.93	1.37
0.923	1.4785	184.6	169.5	0.92	1.34
0.922	1.4788	183.1	168.6	0.59	1.32
0.923	1.4788	184.6	169.0	0.64	1.35
0.923	1.4788	184.1	168.7	0.63	1.31

Ordinarily it is rather difficult to quickly summarize results of this character when reported in a lengthy table. Accordingly, for the convenience of those who may wish to quickly summarize the results of the analyses, the results given above have been plotted as curves. These curves are reported in chart No. 325 which follows:

In order to make it possible for one to compare the chemical and physical characteristics of the above oils with the official specifications[1] for the chemical and physical characteristics of medicinal oil, the latter data are reported on next page:

[1] The Pharmacopœia of the United States—Tenth Revision. J. B. Lippincott Company, Philadelphia, Pa. Page 263.

Graphic representation of variations in the chemical characteristics of 111 *samples of cod liver oil*

CHEMICAL AND PHYSICAL CHARACTERISTICS OF MEDICINAL COD LIVER OIL

Sp. Gr.	Ref. Ind.	Sapon. Value	Iodine Number	F. F. A. Per cent	Unsapon. Per cent
0.918-0.927	Not reported	180-190	140-180	1.41	1.5

Comparing these values with those obtained in the analysis of the samples discussed here, it will be noted that with the exception of one or two minor deviations, all the samples in question met the specifications of the U. S. Pharmacopœia. Nevertheless, it is interesting to note the extent to which the chemical and physical characteristics of the different samples vary. The minimum and maximum specific gravities obtained for the oils in question were 0.920 and 0.927 respectively. The refractive index of the samples varied from 1.4772 to 1.482. The saponification number of the different oils varied from 180.8 to 190.8 and within these limits the different samples varied to such an extent that few samples had the same saponification number. The iodine values varied from 147.5 to 183.1, but within these limits the iodine values were somewhat more constant than was the case with the saponification numbers. The free fatty acid content of the oils in question varied considerably for the first few samples analyzed. Only four samples exceeded a fatty acid content of 1.25%. The majority of the samples had a free fatty acid content under 0.75% and approximately one third of the total of samples had free fatty acid content of less than 0.3%.

In general, the unsaponifiable content of the oils under consideration was between 1.0% and 1.25%. A comparison of the analyses of the oils shows that the percent of unsaponifiable matter in the oils was more nearly uniform than the values obtained for specific gravity, refractive index, saponification value, iodine number, or free fatty acid content.

As one considers these results the question naturally arises as to the cause for the variation in the different oils. Without doubt, many factors contribute to this condition, but in the absence of evidence to the contrary one may assume that the nature of the diet of the fish probably very materially influenced the characteristics of the liver oils. Numerous investigators, such as Shutt[4], Richardson[5], Rosenfield[6], and Mendel and Anderson[7], have shown that the nature of the diet of land animals has a profound influence on the characteristics of the body fat. Furth[8] claims that the characteristics of human fat are influenced by the nature of the diet. As evidence, he states that the skin fat of babies fed breast milk is richer in unsaturated fatty acids than that of babies fed artificial milk.

The conclusion to be drawn from these results is that the chemical and physical characteristics as well as the vitamin potency vary with different cod liver oils. Accordingly, investigators using cod liver oil as a subject of investigation and others who interpret the results of cod liver oil investigations should remember that cod liver oil can not be considered as a chemically pure substance with uniform characteristics and vitamin content. Instead it should be remembered that any cod liver oil obtained in the open market will in all probability have somewhat different chemical and physical characteristics and vitamin content than those of another cod liver oil obtained in the open market.

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[4] Canada Expt. Farms Repts. (1889) pp. 151-155; Idem. Bull. 38 (1901).

[5] Jour. Amer. Chem. Soc., Vol. 26 (1904) No. 4, pp. 372-374.

[6] Ergebn. d. Physiol., Vol. 1 (1902) p. 676.

[7] Oil & Fat Industries, Vol. III, No. 11, Nov., 1926, p. 396.

[8] Problems of Physiol. & Pathological Chem. of Metabolism (1916) p. 383, Lippincott Co., Phila. and London.

Chemical Aspects Of Mayonnaise

An Address Before the Mayonnaise Products Manufacturers Association

BY D. M. GRAY*[1]

MAYONNAISE is seldom thought of as possessing many chemical features but a rather hasty analysis of its composition and behavior discloses the fact that it is closely linked with the chemical, and that chemistry and chemical engineering can be of great service in explaining certain features of its behavior and in overcoming certain difficulties.

In this paper, a few of these chemical aspects will be discussed briefly. To cover fully all the chemical features of mayonnaise and its constituents would be a very large undertaking and the findings would require many times the space required for this paper.

Mayonnaise dressing belongs to that class of substances known as emulsions. There are two general types of emulsions, water-in-oil and oil-in-water. It is with emulsions of the oil-in-water type that we are chiefly concerned as mayonnaise dressing belongs to this type.

An emulsion is a rather peculiar body, because while it is generally quite uniform, one of the components is in a finely divided condition while the other component is undivided or continuous and surrounds completely all of the divided portion.

For the sake of future reference we will refer to the divided portion as the divided phase and to the undivided portion as the continuous phase.

In an oil-in-water emulsion (like mayonnaise) the oil is broken up into droplets, hence it is the divided phase, while the water is unbroken, so it is the continuous phase. In a water-in-oil emulsion the water is the divided phase and the oil the continuous.

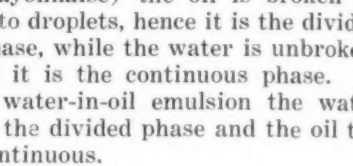


Plate 1

Graphic representation of two contrasting types of emulsions

Plate 1 is a diagrammatic representation of the two common types of emulsions. On the left, the divided drops of water are shown suspended in and surrounded by oil. For this reason, we refer to this type of emulsion as the *Water-in-oil.*

On the right is the commoner type, that of *oil-in-water* in which we are particularly interested on account of mayonnaise. In this type you will note that the drops of oil (the divided phase) are suspended in and surrounded by water (the continuous phase).

Mayonnaise dressing, milk, vanishing cream, and many pharmaceutical emulsions (cod-liver oil,

*Chemical Engineer, Hazel Atlas Glass Co.

[1] *Reprinted by permission from "The Spice Mill."*

for instance) are examples of oil-in-water emulsions. Cold cream, certain petroleum emulsions, emulsions of water with transformer oils are examples of the water-in-oil types of emulsions.

It is seldom important to determine to which type a given emulsion belongs but it can be done quite readily when necessary. One method is by testing whether or not the emulsion will conduct electricity. If it does conduct it, it is of the oil-in-water type. If not, then it is of the water-in-oil type.

In making most all emulsions, a third substance is needed, the emulsifying agent. The number of emulsifying agents is almost legion. Egg yolk and white, gum tragacanth, gum acacia, Irish Moss, casein and dextrin are the commoner, while saponin, hæmoglobin, lacmoid, pepsin, peptone, dextrine, and soap, are good emulsifying agents, though not suitable for food products. On the emulsifying agent depends for the most part, the type of emulsion that will be formed, as will be brought out in a moment.

Mechanism of Emulsification

The mechanism or process of emulsification is as follows: The emulsifying agent with the amount of water necessary to dissolve it or more water if desired is in the mixer or agitator. As the oil is added slowly with stirring it is broken up into droplets. These droplets immediately become coated with a film of the emulsifying agent—egg yolk or whatever is used. When two or more droplets touch together they remain as separate droplets, rather than uniting to form a larger drop, because they are separated by the film of egg yolk enveloping them. This film, to be sure, is exceedingly thin but nevertheless is very effective in preventing the union of the drops, unless damaged in some way, as will be discussed a little later.

In short, the whole idea in emulsification is to get the oil, or divided phase, broken up into as many fine droplets as possible, each droplet being coated with a film of the emulsifying agent.

An interesting point to consider here is the effect of a continuous and very vigorous agitation as compared to an intermittent or less violent agitation. The too vigorous agitation defeats its purpose because not only the oil but also the water is broken up into fine droplets, so that the water phase, which must be continuous, becomes a divided phase, like the oil, so that a proper emulsion can not be formed. Under these conditions the oil droplets reunite after being broken up because they are not properly coated with egg or emulsifier, since this emulsifier is dissolved in the water portion.

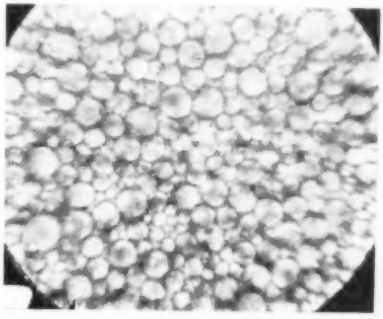


Plate 2
Mayonnaise (enlarged one thousand diameters)

By means of intermittent agitation where there is a short pause between periods of agitation, the water droplets have a chance to reunite, as there is no film holding them apart, but the oil droplets

can reunite only very gradually.

Plate 2 is an actual photomicrograph in which the mayonnaise has been magnified approximately one thousand diameters. When projected on the screen it is further enlarged about twenty-five times, making the image of the particle on the screen about twenty-five thousand times the size of the particles as they actually exist in mayonnaise.

In all the following plates excepting, of course, the charts and photographs of mayonnaise samples exposed to air, the magnification is the same. You will note how the oil is in the form of small droplets, each of which is spherical in shape and is enveloped by a film of egg yolk. It is this film which prevents the reuniting of the droplets and causes the emulsion to be permanent. Each droplet is separated from its neighbors by the continuous water phase. It is this water phase which contains the acid of the vinegar or lemon juice, the salt, sugar and other ingredients not soluble in oil. The egg or other emulsifying agent is distributed in this water portion.

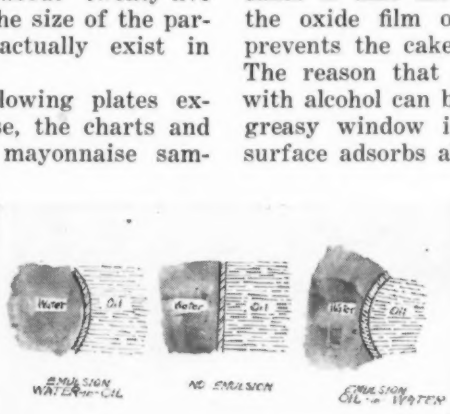


Plate 3
Diagrammatic illustration of action of emulsifying agents

In connection with the formation of mayonnaise emulsion there is a process which physical chemists call *adsorption*, not absorption. This covers a multitude of sins but really has some merits.

Adsorption is a concentrating or condensing of one substance on the surface of another substance but when the first substance penetrates or soaks into the interior of the second, that is absorption. A sponge may absorb water, that is, the water soaks into the interior, but a piece of glass may only adsorb the water on its surface. The reason that an aluminum griddle does not require grease in making cakes is that the air adsorbed by the oxide film on the aluminum prevents the cakes from sticking.[1] The reason that a rag moistened with alcohol can be used to clean a greasy window is that the glass surface adsorbs alcohol more readily than grease so that the grease is displaced even though it does not dissolve in the alcohol. Now, physical chemists claim that the egg is absorbed on the surface of the oil droplets in a mayonnaise emulsion. The egg does not dissolve in the oil drops but condenses or concentrates on the surface to such an extent that a protecting film is formed around the oil droplet which effectually prevents its reuniting with the other droplets.

Certain substances added to mayonnaise have a tendency to concentrate more egg on the surface of the oil or, chemically speaking, they increase the adsorption of the egg on the oil. This causes the emulsion to be thicker, and more permanent. Salt is an example of this class. Other substances decrease the adsorption (that is they decrease the concentration of the egg film on the oil). This makes the

[1] Bancroft, "Applied Colloid Chem.," p. 82.

emulsion thinner or may break it down completely. Alum is an example of this class. More will be said of this later.

Mention was made above that the emulsifying agent had a lot to do with determining whether an emulsion of water-in-oil or one of oil-in-water would be formed. In general, emulsifying agents which are soluble in water, are adsorbed on the surface of the oil and therefore form an emulsion of oil-in-water like mayonnaise. Examples of this class are egg yolk, gum acacia, gum tragacanth, dextrin, casein, soap and saponin.

Agents which are soluble in oil, are adsorbed on the surface of the water and therefore form emulsions of water-in-oil. Examples of this class are calcium oleate, rosin, and magnesium stearate.

Plate 3 shows diagrammatically how the film of emulsifying agent bends around the droplet which it encloses. On the right—illustrating a portion of a drop of oil in an oil-in-water emulsion—the egg is adsorbed on the surface of the oil droplet, and bends around it. When this adsorption is *increased* the bending tendency is *increased*. When it is *decreased*, the bending tendency is also *decreased*. If decreased sufficiently, a condition of "no emulsion" may be reached as shown in the center. Under these conditions the egg film does not remain intact and the emulsion is broken down.

On the left, is shown a film of emulsifying agent—not egg—but one which is adsorbed on the surface of the water and is bending around the droplet of water in a water-in-oil emulsion. Such emulsifying agents are generally soluble in oil rather than water.

If substances are added to this emulsion which *increase* the adsorption of the emulsifying agent on the surface of the water, the film of emulsifying agent tends to bend more. If substances are added which *decrease* the adsorption the tendency to bend is reduced and may even reach the neutral form shown in the center.

Ingredients of Mayonnaise

For the sake of clearness, let us note the ingredients of an average mayonnaise as there are a number of points with respect to each one that should be brought out.

First, comes the oil. It may be any of the edible oils—corn, cottonseed, peanut, sesame, and others. More will be said of these oils later under the subject of Discoloration.

Next comes the emulsifying agent, egg yolk generally, or perhaps the whole egg. These have already been discussed at some length.

Then thickening agents such as starch or flour which are sometimes added. There is a word or so to be said about these under the subject of Breaking Down of Mayonnaise.

Along with this, we have the seasoning ingredients, salt, pepper, paprika, sugar, mustard, etc., which are of interest in Discoloration.

And last, we have the acid ingredients which bring most of the water content into mayonnaise. Generally, the acid is in the form of vinegar but sometimes lemon juice or citric, acetic or lactic acids are used. The effect of adding the acid ingredients is to thin down the mayonnaise to a considerable extent. The acid with its accompanying water, used in the usual amounts, does not cause the mayonnaise to partly break down, it merely flows

in between the droplets of oil causing them to be at greater distances from each other. The result is that the mayonnaise is much thinner in consistency.

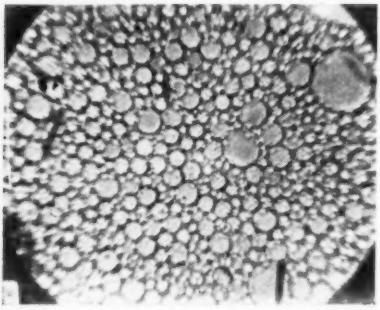


Plate 4
Heavy mayonnaise emulsion (enlarged one thousand diameters)

Plate 4 is a mayonnaise emulsion with only a small amount of water. Plate 5 shows this same emulsion after it has been thinned with vinegar. You will notice that the size of the individual particles is no greater, they are only farther apart, showing that the addition of the usual amount of vinegar does not break down the emulsion—it merely makes it more fluid by flowing in between the oil droplets. The reason that an unthinned mayonnaise is so stiff is that the oil droplets, covered with the film of egg, rub one another closely giving them a tendency to "hang together." When a little more vinegar or water is added it naturally lessens this "hanging together" tendency by flowing in between the droplets, with the result that a great reduction in consistency takes place.

One of the afflictions to which most all mayonnaise is susceptible is that of separation or breaking down, also called demulsification. This may come from one or several of a number of different causes and it is one of the purposes of this paper to outline some of the causes, their chemical explanation, and possible cures.

The first cause which we might take is that of the wrong proportion of oil, water, and egg or emulsifying agent. For a permanent emulsion of these three ingredients there seems to be a certain definite proportion for each one, which can be varied only between fairly narrow limits and still maintain a permanent emulsion. For instance, in a certain set of experiments with 78 per cent of oil, 17 per cent of water and 5 per cent egg yolk, a permanent emulsion was formed. If the amount of oil was reduced

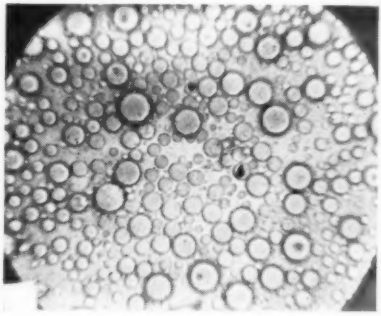


Plate 5
Emulsion shown in Plate 4, *after thinning with vinegar*

to, say, 76 per cent, the water increased to 19 per cent, the egg remaining the same, on standing the emulsion separated into two layers —a watery layer below and a creamy layer of emulsion above. If the percentage of oil was increased to 92 per cent, the egg remaining the same, the emulsion also separated on standing but this time with an oil layer above and the emulsion layer below. If the amount of oil was decreased be-

low 78 per cent and the water increased, but the egg yolk increased also, the emulsion formed was permanent. In general, the more water in an oil-and-water emulsion the more emulsifying agent is needed to make it permanent. Egg yolk was also found to have a greater emulsifying power than the whole egg.

It is impossible to give exact or

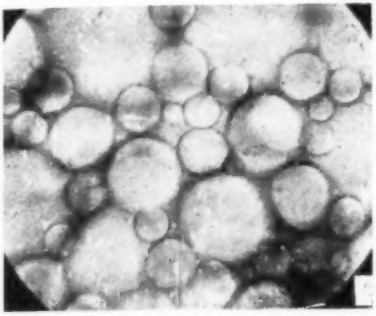


Plate 6
Mayonnaise in which oil has been added too rapidly (× 1000)

even approximate ratios of oil, water and egg which will hold for all kinds and mixtures of mayonnaise because the kind of oil, the condition of the egg, or other emulsifying agent, the presence or absence and character of thickening materials, if any are used, and many other factors make each case a special one for which no general rules can be given.

The second cause of breakdown is that due to too rapid addition of oil. Under such conditions the oil is added more rapidly than it can be broken into small droplets and coated with a film of emulsifier. The result is that the emulsion is coarse, that is, the oil droplets are large and when several of them come together they have a tendency to unite and form still larger droplets, so that we get separation of oil or at least a dressing of poor consistency.

Plate 6 shows the effect of adding oil too rapidly. The drops of oil are too large. The oil has not been properly broken into smaller droplets. The result is a dressing of thin consistency and of poor permanence.

The third cause is using the wrong kind of agitation. As mentioned above too violent an agitation is almost worse than none because it has a tendency to break up the water into droplets as well as the oil, and to prevent the water droplets from reuniting which, of course, is fatal to a good emulsion. It is scarcely worth while to do more than mention this third cause, because of the number of machines on the market which are designed especially to give the correct agitation and with which all manufacturers of mayonnaise are much more familiar than is the writer.

The fourth cause with which we are concerned is the breakdown of an emulsion due to heat. It is a well known fact that when liquids (like gases and solids) are warmed they expand and when cooled they contract. The amount of this expansion or contraction for a fixed change in temperature is called the *coefficient of expansion* and differs for different liquids. The coefficient for a certain edible oil is greater than that for water (.000721 oil and .000207 water),[1] which means that for a given change in temperature the oil will either expand or contract *more* than the water will. This causes forces to be set up in the emulsion which break the protecting egg film and force the oil droplets to unite,

[1] Fowle 'Smithsonian Physical Tables," 1923, p. 221.

thus breaking down the emulsion. The greater this change in temperature, the greater will be the forces set up and the greater the tendency of the emulsion to separate. The finer the droplets of oil in an emulsion and the better they are

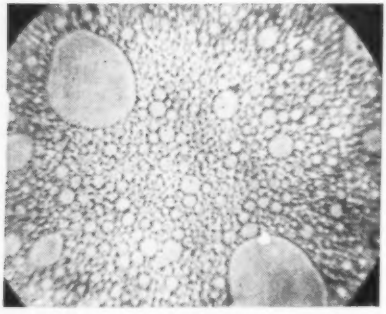


Plate 7
Mayonnaise before heating

coated with egg or emulsifier, the less is the tendency of the emulsion to break down when subjected to severe temperature changes.

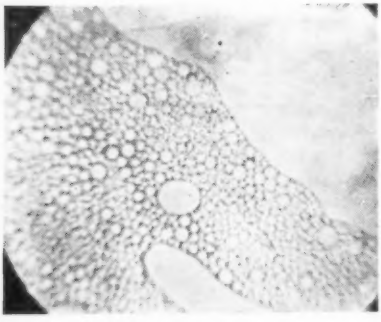


Plate 8
Same mayonnaise after slight heating

Plates 7, 8, and 9 show the effect of heat just referred to. Plate 7 is the mayonnaise at the start. The oil droplets are small, although there are several large droplets. Plate 8 shows the effect of heat, a patch of oil forming by the breaking down of the small droplets. Plate 9, taken a few minutes later on the same specimen, shows that the breakdown is increasing, as evidenced by the increased size of the oil patch.

The fifth cause of breakdown with which we have to deal is freezing. The remarks concerning the unequal expansion of oil and water which takes place during a rise in temperature apply with equal force when the temperature is reduced. When water is cooled, say, from room temperature down to 39 deg. F., it contracts. On being cooled still lower it ceases to contract and actually begins to expand so that ice is less dense than water, which is attested to by the fact that ice floats in water. Following the course of events, when mayonnaise is cooled strongly we see that the oil droplets contract to a greater degree than does the water surrounding them. This alone has a tendency to break up the emulsion. After the emulsion reaches 39 deg. F. (7 deg. above freezing) the water begins to expand. This aggravates the state of affairs greatly, so that by the time the freezing point is reached the emulsion is apt to be completely broken. When the mixture thaws the oil and water seem to have changed places, as oil surrounds fairly large patches or drops of water. In these drops, in turn, may be seen more droplets of oil. In other words, we practically have an emulsion of oil-in-water within an emulsion of water-in-oil. The mixture, of course, does not have the outward appearance of normal mayonnaise because of the separation of the oil.

Plates 10 to 14 show what hap-

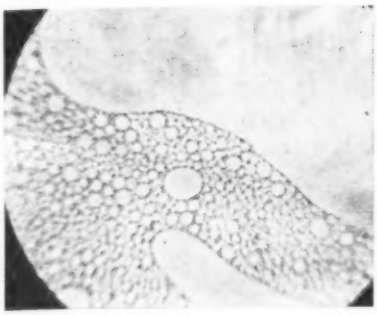


Plate 9
Mayonnaise after considerable heating

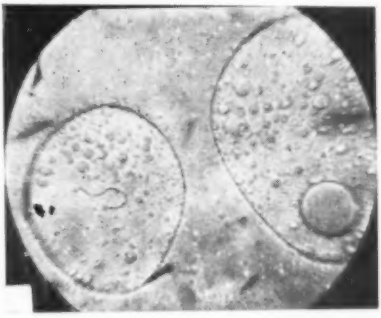


Plate 12
Frozen mayonnaise

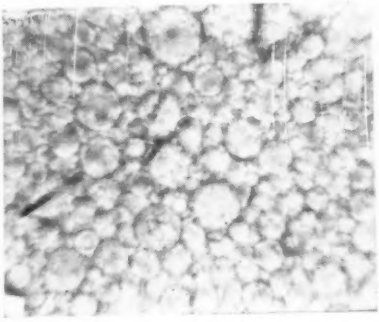


Plate 10
Mayonnaise before freezing
(× 1000)

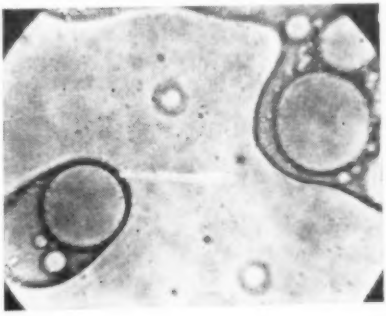


Plate 13
Frozen mayonnaise (another view)

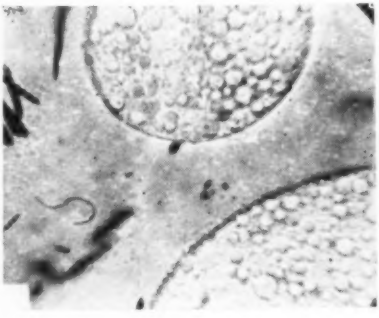


Plate 11
Same mayonnaise when frozen

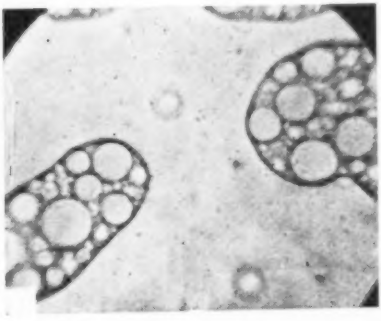


Plate 14
Frozen mayonnaise (another view)

pened when a sample of mayonnaise was frozen. Plate 10 shows the original condition before being subjected to the cold. Plate 11 shows the sample after being frozen. The plate shows that the oil surrounds drops or patches of water. Within easy reach of these patches can be seen small droplets of oil. The appearance of the whole sample reminds one of Schweitzer cheese. The cheese proper corresponds to the oil, while the gas holes in the cheese correspond to the patches of water. Plates 12, 13 and 14 are different views of the same sample. An interesting point to note here is that this peculiar appearance was noticeable in the frozen sample and as it thawed out there was scarcely any change in appearance.

Chemical Causes of Separation

The next group of causes of the breaking of the mayonnaise emulsion we may classify as strictly chemical causes. During the course of an investigation as to the reasons why a certain mixture of mayonnaise and relish broke down, it was discovered that the mayonnaise stood up well until mixed with the pickle relish. As alum causes breaking down of certain emulsions and as alum is very frequently used in pickles, the relish was examined for alum and it was found to be present. The effect of alum in mayonnaise dressing was studied very carefully under the microscope. The accompanying plates show very clearly what takes place.

Plate 15 shows the orginal mayonnaise. Plate 16 shows a solution of alum flowing into this same mayonnaise. The blurred portion is due to the moving current of alum solution, carrying along with it a number of oil droplets. You will notice that breaking down of the emulsion has already begun, as evidenced by the formation of several large oil drops. The flow of alum solution into the sample was secured by placing the end of a small strip of absorbent paper under one edge of the cover glass, which is a small disc of very thin glass covering the mayonnaise under examination. The alum solution was placed on the other end of the paper and was thereby drawn under the cover glass and distrib-

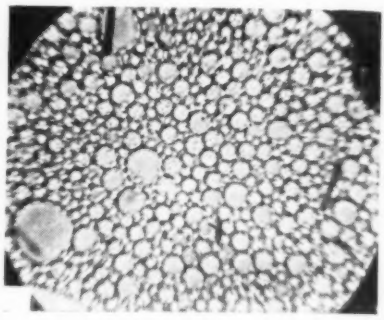


Plate 15
Mayonnaise before addition of alum
(× 1000)

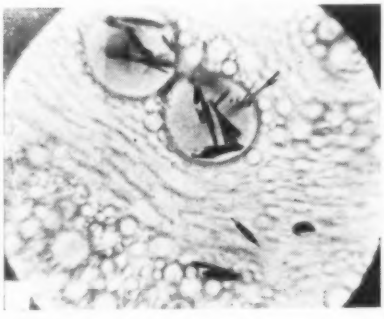


Plate 16
Alum flowing into mayonnaise emulsion

uted throughout the mayonnaise sample. Plate 18 shows, in the same manner, the effects the alum has brought about in breaking down the emulsion, that is, in causing the small droplets of oil to unite and form larger ones.

The peculiar particles seen in the oil drops in plates 16 and 18 are crystals of an oil soluble dye. This dye was dissolved in the oil to give it a color contrasting with

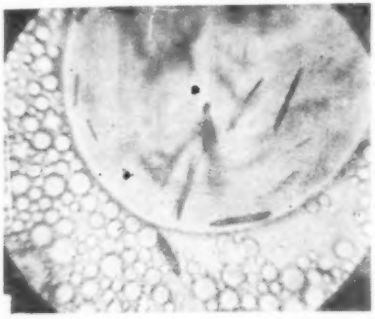


Plate 18
Mayonnaise emulsion separated by addition of alum

the water portion to give better results in photographing. Some of this dye crystallized in the oil as shown, but this does not affect the behavior of the emulsion.

Plate 21 shows the emulsion about to reach the neutral point. The drops are seen to be losing their spherical shape and are forming in irregular masses. Plate 22 shows that the neutral stage has been reached. In other words, the emulsion has ceased to be an emulsion—it is a mixture in which the oil and water are intermingled in the form of large and irregular patches.

The effect of salt is just opposite to that of alum. When a little salt is added to mayonnaise it causes a very noticeable thickening. This

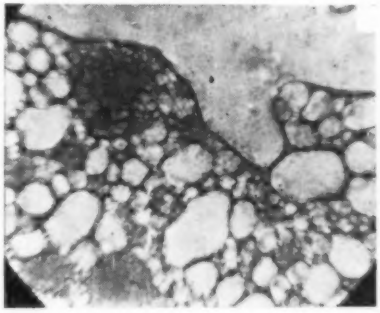


Plate 21
Same emulsion at point of complete separation

thickening is found to be due to the droplets of oil having become more completely emulsified.

Plate 23 shows a mayonnaise which has been purposely made up in the form of a coarse emulsion by adding the oil rapidly during mixing. After all ingredients were added it was beaten five minutes longer so that if the emulsion was going to become finer it would have an opportunity to do this before the salt was added. About 1 per cent of salt was then added, the mixture beaten a few seconds to thoroughly incorporate the salt, with the result shown in plate 24. The effect of the salt

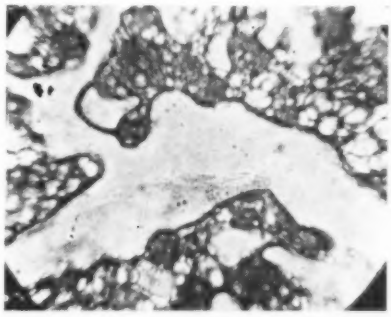


Plate 22
Same emulsion entirely separated

was to cause the formation of a much finer and more permanent emulsion, as evidenced by the reduction in the size of the oil droplets. This reduction in size explains why the emulsion became so much stiffer in consistency—because the smaller droplets can get closer together and come nearer at more points. In general, the whole structure is more closely knit and therefore stiffer in consistency.

The question naturally arises. "Why does alum cause this breaking down while salt causes thickening?" For the explanation we must again refer to adsorption—that phenomenon which takes place where the surface of the oil droplet and the surface of egg solution in water, meet. Common salt is a compound containing the element sodium. This sodium carries *one* charge of positive electricity. Alum is a compound containing the element aluminum. This aluminum carries *three* charges of positive electricity. Now, a little bit of positive electricity does not hurt an emulsion; in fact, it helps make it more stable, as we have seen above, with common salt, but when very much positive electricity gets into the emulsion it has a great tendency to break down the emulsion and cause the separation of the oil.

The reason for this is that the charges of positive electricity are adsorbed at the surface between the oil and egg and have a tendency to break down the protecting film around each oil droplet. The more charges of positive electricity adsorbed at the surface, the more the emulsion will break down.

Plate 24-A illustrates the relative amounts of different chlorides required to break down an emulsion of oil-in-water to the same extent.[1] As we saw before, a small amount of salt (sodium chloride) was beneficial to an emulsion of oil-in-water. However, if the amount of salt added is very great it will have a tendency to break down the emulsion. Needless to say, long before this much salt is added the extreme saltiness of the mayonnaise would render it unfit for consumption. We note from the chart that it takes approximately 300,000 parts of salt to equal 6,000

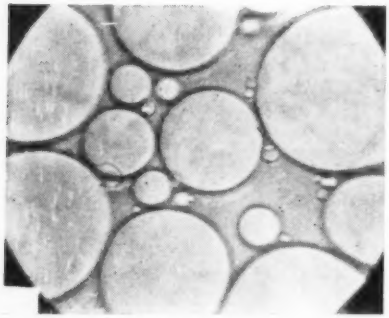


Plate 23
Mayonnaise with heavy oil-drops due to rapid mixing (× 1000)

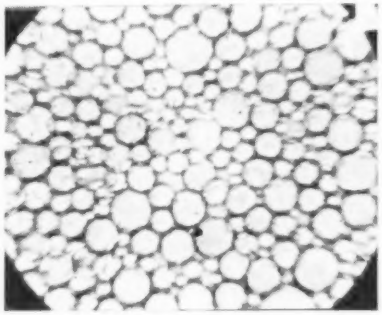


Plate 24
Same mayonnaise after incorporation of 1% *of salt* (× 1000)

[1] Derived from table due to Powis-Clayton, "The Theory of Emulsions and Emulsification," p. 26 (1923).

parts of calcium chloride or 33 parts of aluminum chloride (similar chemically in many respects to alum) or 1 part of thallium chloride. In other words, thallium chloride, with its four charges of positive electricity attached to the thallium, is the most potent in breaking down the emulsion, while

ABILITY OF CHLORIDES TO "BREAK" AN OIL IN WATER EMULSION.

CHLORIDE	FORMULA	RELATIVE AMOUNT REQUIRED TO BREAK EMULSION
SODIUM CHLORIDE	$Na^{+}Cl^{-}$	320,000
CALCIUM CHLORIDE	$Ca^{++}Cl_2^{-}$	6085
ALUMINUM CHLORIDE	$Al^{+++}Cl_3^{-}$	33
THALLIUM CHLORIDE	$Tl^{+++}Cl_3^{-}$	1

Plate 24a

Showing effect of relative positive valency on action of metallic chlorides on oil in water emulsions

salt (sodium chloride), with its single charge of positive electricity attached to the sodium, is least potent; in fact, just one three hundred and twenty thousandths as effective.

Of more practical interest is the relationship between the sodium and the aluminum compounds. From these figures we may properly expect alum to be approximately ten thousand times as powerful in breaking down an emulsion as is common salt. This sheds light on the question of the small amount of alum in pickle relish exerting such a strong breaking down effect on mayonnaise when mixed with it. In general, with groups of compounds wherein the metals show an increasing number of positive charges we may expect a greatly increased "breaking" effect on oil-in-water emulsions.

Another cause contributing to the breakdown of mayonnaise is that which may occur when thickening agents are used, such as starch or flour. This is due to the action of the acid in the mayonnaise on the starch or flour and is known as "hydrolysis." One of the best known commercial examples of this type of hydrolysis is the manufacture of corn syrup or "glucose," where corn starch is treated with dilute acid which converts it into the sugar, "glucose."

The immediate effect of this hydrolysis of the starch or flour in mayonnaise is to make the dressing much thinner and watery, since a sugar solution is thinner than a starch or flour solution, both having the same water content. If the starch or flour is depended upon to keep the oil emulsified the emulsion is apt to break down, with separation of the oil. In any event, the effect is apt to be bad, because when a manufacturer makes up a dressing of the proper consistency he wants it to be that same consistency when used by the ultimate consumer.

Another cause of breakdown along somewhat the same lines is that due to the spoilage or decomposition of the emulsifying agent. Certain emulsifying agents have a tendency to decompose rapidly, consequently losing their emulsifying power, so that the emulsion breaks down. The remedy here is to use a different emulsifying agent or add an artificial preservative to keep the emulsifying agent from decomposing.

Another cause of separation of oil is due to the evaporation of the moisture from the mayonnaise. As the moisture evaporates from the surface, the protecting egg or emulsifier film dries out also, becomes brittle and breaks, allowing the oil droplets to flow together so that a layer of oil forms on the

surface of the mayonnaise. Separation of oil from this cause is easily overcome, of course, by using such a closure on the mayonnaise that evaporation is prevented.

A cause of breaking down of emulsions which is seldom considered is vibration. An emulsion is a peculiarly constructed body and for that reason is sensitive to unusual influences. Ayres[1] mentions an instance where an emulsion (not mayonnaise, however) was subjected to heating, freezing and centrifuging but could not be broken down. A short ride in an express train caused it to separate completely. This is merely mentioned as a matter of interest and might be useful when all other possible reasons in a case of separation have been looked into.

The question is often asked, "Why is it so difficult if not altogether impossible to re-emulsify mayonnaise which has separated?" If the emulsion has been broken by heat, the chances are that the egg has been changed so that it does not have its original emulsifying power. If by freezing then the egg is not changed after it thaws out but the oil is distributed in large patches. If the oil, egg, and other ingredients could be completely separated, and then remixed in the usual manner, adding the oil slowly, a normal mayonnaise could doubtless be made. The same applies to breakdowns due to too rapid addition of oil, improper agitation, and vibration. If the breakdown is due to alum or similar compounds, they would have to be removed before a permanent emulsion could be formed.

If due to hydrolysis or decomposition of the emulsifying agent, it cannot be re-emulsified unless more emulsifying agent is added and a new start made.

[1] J. S. C. I. (1916), 35, 678.

Discoloration

The next subject with which we have to deal is Discoloration or Darkening. Very little is known about the actual causes of discoloration. Certain brands of mayonnaise darken rapidly when exposed to air, while others show only a slight discoloration. As this darkening or discoloration occurs only on the surface which is actually exposed to the air, it must be due to some action of the air on the product. The most active element in the air is oxygen, so that one would naturally attribute the darkening action of the air to the oxygen which it contains.

It is a well-known fact that certain oils absorb oxygen from the air. In other words they oxidize. In this process they change from their oily nature into a tough leathery film. They are said to "dry" and oils which have this property to a pronounced degree are called drying oils. Linseed is the most familar example of this class. Oils which have this property in a lesser degree are called semi-drying oils, and those which do not show any appreciable drying are placed in the non-drying class. There is no sharp distinction between the classes.

Plate 25 is a chart showing the amount of oxygen which various oils absorb under the same set of conditions. Linseed, of course, heads the list with an oxygen absorption of 16 per cent. Next comes corn oil with about 10.6 per cent, cottonseed with about 9 per cent, sesame 8 per cent, peanut 6.5 per cent, olive 5.25 per cent and paraffin oil practically zero.

Bear these relative amounts in mind, as we will refer to them in just a moment. Neither linseed nor paraffin oil are, of course, edible, but are given here by way of comparison as they represent the two extremes.

Samples of mayonnaise dressing were made up alike in all respects except that a different oil was used in each. Corn, cottonseed, peanut, olive and paraffin oil were used. These samples were exposed to the air in shallow glass dishes and the discoloration noted after 24 and 48 hours.

Plate 26 shows one of these shallow glass dishes which has been filled with mayonnaise and exposed to the air. In the plate the discolored portion shows black, but, of course, this actually was a dark yellowish brown. The light spot in the center is the portion of mayonnaise which was covered at the outset of the test with a very thin cover glass. This protects the surface of the mayonnaise immediately under it from the action of the

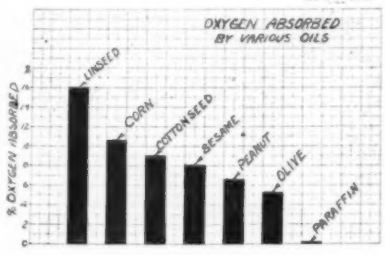


Plate 25
Relative oxygen absorption of various oils

air and as it prevents this portion from discoloring, serves by way of comparison to tell at a glance just how much discoloration has taken place on the exposed surface.

The examination shows that the extent of darkening is in proportion to the oxygen absorption of the oil. In other words, the more oxygen that the oil has a tendency to absorb the greater will be the dis-

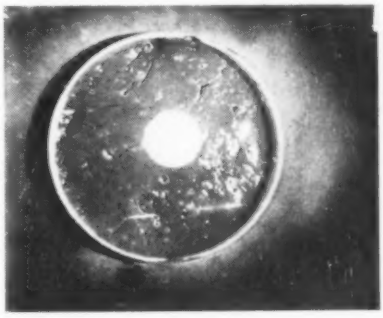


Plate 26
Effect of air-exposure in darkening mayonnaise

coloration of the mayonnaise made from that oil.

Plate 27 shows the relative darkening of the samples made up with the different oils. The oils used constitute the only differences between the respective samples, so that any differences in discoloration must be due to different behavior of the respective oils. The sample made with corn oil shows most darkening, cottonseed next, peanut next, and paraffin oil least. By referring again to Plate 25, we see that this is in the order of decreasing oxygen absorptions. In other words, this test showed in general that the higher the oxygen absorption value of an oil, the greater the darkening or discoloration of the mayonnaise made from it when exposed to air. Certain other factors influence the darkening as will be noted later but the relation of oxygen absorption to darkening is of fundamental importance and should not be lost sight of.

Paraffin oil is practically non-oxygen absorbing so that we shall expect it to show the least discoloration and the experimental facts prove this to be correct. Paraffin oil is used here because it *is* non-

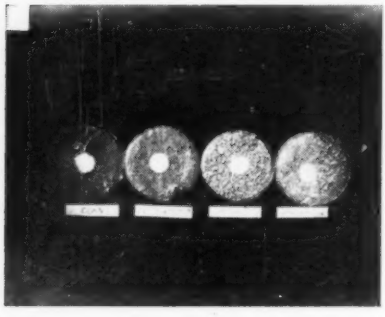


Plate 27
Comparative darkening under air-exposure of mayonnaise mixtures made from various oils

oxygen absorbing, and serves as a check on the other oils. Inasmuch as even *some* discoloration occurs on mayonnaise made with paraffin oil, although the discoloration is least of all the oils, there must be a factor besides the oxygen absorption of the oils. Therefore, it must be true that some of the discoloration is brought about by exposure of the egg to the air.

Some experiments were then undertaken to determine the effect, if any, of the other ingredients on discoloration. By other ingredients we refer to the ingredients beside oil, egg, and water; namely, salt, vinegar, mustard, pepper, paprika, lemon juice, sugar or any other added material.

Samples of mayonnaise were made with cottonseed oil, which shows moderate but not excessive darkening. To different samples were added ordinary vinegar, sterilized vinegar, acetic acid of the same strength as the vinegar used, lemon juice, citric acid of the same strength as the lemon juice used, mustard, pepper, and paprika. Samples were also prepared with and without salt, and with water in the place of vinegar.

The summary of these tests was:

First.—An ordinary amount of salt (1%) does not appear to increase or decrease discoloration.

Second.—Sterilized vinegar gave no improvement over ordinary vinegar. This test was carried out as it was thought possible some of the darkening might be due to bacteria or other organisms in the vinegar.

Third.—Samples made with acetic acid of the same acid strength as vinegar were found to discolor less than those made with vinegar.

Fourth.—Citric acid showed a very slight improvement over lemon juice but neither lemon juice nor citric acid were as good as acetic acid.

Fifth.—With samples containing *mustard* and vinegar the discoloration was much greater than with vinegar only.

Sixth.—Pepper and paprika were found to exert a darkening effect. Of these two, paprika was the most noticeable.

Summing up we would say that some benefit could be secured by substituting acetic acid for vinegar; that mustard appears to be responsible for a considerable amount of darkening and to a lesser extent pepper and paprika; that a moderate amount of salt has no effect.

Just why mustard and spices present in the small amount that they are should cause such a considerable darkening is hard to explain. A possible explanation is

that the powerful volatile and non-volatile oils which these substances contain act like driers on the oil and cause them to absorb more oxygen than they otherwise would and therefore cause them to darken to a greater extent. This action would be similar to that occurring when a small amount of drier (approximately 1%) is added to linseed oil in making paint. These driers (generally compounds of lead, manganese or cobalt) cause the linseed oil to absorb oxygen very rapidly, and the paint dries in about 24 hours whereas if the drier were not used, the paint would still be soft at the end of several weeks.

In considering the whole subject of discoloration, one is impressed with the fact that the changes which take place are very complex and appear to be a combination of factors rather than any one. The oxidation of the oils is one thing, however, that is fairly well established, so that in suggesting ways and means of eliminating or at least reducing discoloration, the use of non-drying oils or oils of low oxygen absorption, would be one means.

A more feasible way commercially is to fill the package quite full so that the amount of air space above the mayonnaise is reduced to a minimum, thus reducing the amount of oxygen which the oil can absorb.

Another is to seal the mayonnaise under vacuum. Samples of vacuum packed mayonnaise have been found to show an almost negligible discoloration.

And finally, the careful selection of spices and a sparing use of them and of mustard particularly should give considerable relief from discoloration.

Special Notice A. O. C. S.

The Research Associate of the Society, Miss Walker, at the Bureau of Standards, Washington, will probably be ready to standardize all red glasses January 2. Make your arrangements with A. W. Putland, Portsmouth Cotton Oil Refining Corporation, Portsmouth, Va., for dates when the glasses can be submitted to avoid unnecessary delays. The charge for each glass will be $1.50.

In connection with this matter, the Governing Board has adopted rules requiring all referee chemists to have the following certified glasses as a minimum standard set: 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1.0, 2.0, 3.0, 3.5, 4.0, 5.0, 6.0, 7.0, 7.6, 8.0, 9.0, 10.0, 11.0, 12.0, 16.0, 20.0, in the reds, and 1.0, 2.0, 3.0, 5.0, 10.0, 15.0, 20.0, and 35.0, in the yellows. After the necessary time for certification of the glasses has been allowed, referee certificate will be refused unless these glasses are owned by the chemist applying.

Harris Takes Over Parks-Cramer Line

Mr. John P. Harris, Chemical Engineer, of 400 N. Michigan Avenue, Chicago, Ill., who has been representing Industrial Chemical Co. in Western and Southern territory, has added to his lines the indirect oil-heating deodorizing equipment of Parks-Cramer Co. of Boston, Mass., offering the Merrill System of deodorizer heating and control. Mr. Harris will represent the Parks-Cramer Co. in the same territory which he will continue to cover for Industrial Chemical Co.

The Chemical Composition of Spanish Olive Oil

BY GEORGE S. JAMIESON*

THE composition of representative samples of California, Italian, and Tunisian olive oils has been determined previously in this laboratory (OIL & FAT INDUSTRIES II, p. 40 and 110; IV, p. 63). A two-gallon sample of olive oil from the region of Borjas, Spain, which was received two years ago, has recently been examined so that a comparison can be made of the composition of oils obtained from olives grown in different parts of the world. Borjas olive oil is characterized as greenish in color, very sweet, and with a fruity taste; the sample examined having all these properties.

Characteristics

The more important chemical and physical characteristics are recorded in Table I, and for comparison the characteristics of the other three oils also are given. The methods used were the same as those employed for the examination of the other three oils.

TABLE I

Chemical and Physical Characteristics of Olive Oil

Samples	Spanish Oil	Italian Oil	Tunisian Oil	Californian Oil
Specific gravity 25°/25°	0.9116	0.9120	0.9131	0.9119
Refractive index at 20°	1.4689	1.4690	1.4700	1.4690
Acid value	1.8	1.8	1.9	1.5
Iodine number (Hanus)	83.7	84.4	86.0	85.1
Saponification value	192.4	190.8	193.6	190.6
Unsaponifiable matter (%)	0.8*	1.1	0.8	1.0
Saturated acids (det'd) (%)	11.7†	....	....	....
Unsatur. acids + unsapon. (det'd) %	83.4	....	....	....
Iodine No. of unsat. acids + unsapon.	97.8	....	....	....
Iodine No. of unsat. acids.....	97.2	94.2	103.6	94.8
Saturated acids (corrected) (%)	10.7	10.9	16.5	8.9
Unsaturated acids (corrected) (%)	83.6	83.3	77.6	85.2

*Iodine number 157.9.
†Iodine number 8.7.

The iodine number of the unsaturated acids (97.2) shows that this fraction of the fatty acids consists of oleic acid (iodine number 90.1) and linolic acid (iodine number 181.4). The following percentages were calculated, using these numbers.

	Percentage composition of unsat. acid fraction	In original oil	Glycerides in original oil
Oleic acid	92.09	77.0	80.5
Linolic acid	7.91	6.6	6.9

The saturated acids were separated by the lead salt ether method from the oil and were esterified in the usual manner with methyl alcohol and dry hydrochloric acid gas. The methyl esters (119.0 g.)

*Oil, Fat and Wax Laboratory, Bureau of Chemistry and Soils, U. S. Department of Agriculture.

TABLE II

Spanish Olive Oil, Saturated Acids

	Acids in original oil Per cent	Glycerides in original oil Per cent
Myristic	0.19	0.2
Palmitic	8.94	9.4
Stearic	1.34	1.4
Arachidic	.23	0.2

were fractionally distilled under a 4 mm. pressure. The fractions were analyzed, and their compositions were determined as previously described (OIL & FAT INDUSTRIES 2, p. 40).

The results given in Table II were calculated from the analytical data obtained. The acids were recovered from some of the methyl ester fractions by saponifying with alcoholic potash and decomposing the resulting soap with hydrochloric acid. Palmitic, stearic, and arachidic acids were isolated from various fractions by fractional crystallization from alcohol. Their identity was established by their melting points and by observing whether or not these melting points were lowered when the acids were mixed with equal quantities of the respective acids which they were suspected of being, the purity of which had previously been established by elementary analysis. No depression of the melting points was observed in any case.

TABLE III

Composition of Olive Oil

Glycerides of	Spanish oil Per cent	Italian oil Per cent	Tunisian oil Per cent	California oil Per cent
Oleic acid	80.5	83.1	69.1	84.4
Linolic acid	6.9	3.9	12.0	4.6
Myristic acid	0.2	trace	0.1	trace
Palmitic acid	9.4	9.2	14.4	6.9
Stearic acid	1.4	2.0	2.4	2.3
Arachidic acid	0.2	0.2	0.3	0.1
Unsaponifiable matter	0.8	1.1	0.8	1.0

Discussion of the Results

The results given in Table I show that the specific gravities, refractive indices, acid values, saponification values, and the percentages of unsaponifiable matter of the four oils differ but little. With the exception of the Tunisian oil, the percentages of the saturated acids range from 8.9 to 10.9; likewise the iodine numbers of these three oils show a maximum difference of only 1.4. The composition of the four oils in terms of glycerides is given in Table III. An examination of these results shows that there is much similarity in composition of the Californian and Italian oils. There has been considerable difference of opinion in regard to the presence of arachidic acid in olive oils from various localities. It will be observed that these four oils contain from 0.1 to 0.3 per cent of arachidic acid. Tänfel and Sari (Anales soc. espan. fis. quin. 1926, 24, 25) examined a Spanish olive oil but were unable to detect arachidic acid.

Edible and Soap Oil Requirements Mutually Supplementary

Presented Before the First Mid-Year Meeting of the American Oil Chemists' Society

BY MARTIN H. ITTNER*

UNLESS I am greatly mistaken most of our members are primarily interested, either directly or indirectly, in at least some phase of the production of edible oils.

A consideration of the scope of an "oil chemist's" work discloses many interesting relationships. He may be concerned in the yield of oil from a given source and its quality. He is apt to be concerned with refining methods and refining losses: With color, odor and taste and methods of improving these. If he is interested in the economical side of the question as well as the scientific, as he is almost sure to be, he will inquire into the cost of seeds, and he will give heed also to other oil seeds than those he is using. He can not entirely neglect fats from all the various sources, or inedible grades or low grade by-products and the market prices of these. The fact is he is concerned also with soap and glycerine and soap fats and oils that he does not handle.

There was a time when large quantities of cotton seed oil found their way with great regularity into the soap pan. Greater care in refining and education of the public have increased the demand for this oil for edible purposes until its cost is usually well above the cost of fats and oils regularly used for soap-making.

Refining losses send "foots" to soap pans. Foots are made into low grade soap direct by complete saponification and "washing," or into soap powder by mixing with soda ash after saponification, this latter product probably forming their chief outlet. A certain quality of foots is subjected to some form of acid saponification yielding dark colored fatty acids which are then subjected to steam distillation. The fatty acids thus obtained yield a better colored soap than can be made by direct alkaline saponification of foots. There is some loss in this process, however, part of the acids going into a tarry residue and some going into gases.

The edible oil trade and the soap trade are sometimes looked upon as competing for certain oils. In reality this is not the case for the edible oil trade can pay and does pay so much higher prices for oils that the soap trade is forced to look elsewhere or to utilize the poorer grades only, unless there is a temporary overproduction which seldom occurs.

Many of us can remember when coconut oil found little use outside of the soap pan. Now the demand for this oil for use in butter substitutes and other edible products forces the soap maker to pay higher prices or to use poorer grades.

The demand for edible oils is enormous and the need for soap fats and oils is beyond any one's

* Chief Chemist, Colgate & Co.

realization. The demand for each is increasing and will continue to increase.

Oil consumers are often embarrassed by the acts of some legislators who would put prohibitive tariffs on the importation of fats and oils. I believe there has never been a sound basis for such excessive rates nor that there has been a need for it or even a general demand therefor from our producers. The origin of such moves is generally with the legislators themselves who think they see in it a bait to catch more votes.

I would like to see American produced oils so well refined and so superior in quality that there would be a large export demand for them. This is true to some extent now. We should have no prohibitive or even excessive fat or oil duties.

If crude foreign oils can be brought in free, or at low rates, American chemists and American industry can benefit by it. Our chemists and our refineries would then be doing some of the extra business that now goes to foreign countries, our American soap factories would have less trouble in getting their supplies and the public would benefit by cheaper soap.

The oil and fat industry is a multiphase one and no one can do more than touch on a few of these phases in a short talk. I have merely aimed to point out a few of the more important relations that exist in the industry.

Progress in Cotton Oil Milling

A Discussion of Advanced Practice in America's Largest Vegetable Oil Industry

BY ALAN PORTER LEE

JANUARY 1, 1928, will see the cottonseed oil mills of this country well advanced in the season's crush, and the Christmas season is a good time for the miller to take a "breathing spell," and review his situation.

Due to the comparatively short cotton crop this season, we have seen a repetition of that old evil, competitive bidding and overbidding for seed on the part of mill owners or managers, apparently based on the theory of keeping each mill running at any cost.

When the price of oil and meal fails to pay even the cash expended for seed, without considering the operating and interest charges of the mill, there is only one inevitable outcome, the suspension of those mills which lack the needed financial reserve for the absorption of the season's losses.

These conditions are causing more and more mill managers to awaken to the fact that their only salvation lies in sane seed-buying and in the reduction of operating costs in the mill, as interest charges are fixed, and selling costs already so low that they may be considered at an irreducible minimum.

Wages cannot be expected to take any material decline and are more likely to go the other way as the continuing industrial development of the South increases the demand for labor.

The harassed cottonseed oil miller, then must turn to his manufacturing methods as the only source of reductions in his cost of doing business.

He need look no further, for it is safe to say that those mills which are really abreast of the times in modern material handling and processing methods are few and far between.

In most mills on the contrary, there is room for definite savings in every department, from the unloading of seed to the loading of oil in the tank car.

How many mills take their seed from the farmer's wagon or from the box-car by means of a modern pneumatic conveying system instead of with shovels and wheelbarrows or bucket conveyors? The pneumatic conveyor will do the work in half the time at half the power cost of other conveyor systems.

It does not matter whether it is the farmer's time and labor used to unload his seed or not. He will be paid for it in the price of seed, even if he or his farmhand only spends the time sitting in the shade waiting for the wagon to be unloaded, and the seed will always go to the mill with the best unloading facilities, other things being equal.

Having arrived at the mill and been unloaded the seed must be stored.

Careful Seed-Handling Important

How many mills are equipped to properly cool and dry their seed before storing it?

The annual loss to the cottonseed oil millers of this country through the production of off quality oil because of deterioration of seed in storage undoubtedly totals several hundred thousand dollars.

Drying and cooling systems are *not* expensive when the economy of their use is considered.

Now let us step into the linter room. Is it a modern one, with each linter motor-driven, with the seed brought in pneumatically and the lint gathered from each machine and transported to the bailer in the same modern way; or is it an archaic jumble of pulleys, shafting and belting with the air so full of lint that we don't dare stay long if we want to keep on breathing? That lint in the air weighs something and thus a good many pounds of linters are lost in a season. Are there good saw-sharpening machines and are they used regularly, so that the grades of linters produced are uniform and command the best prices?

Good housekeeping in every department of a mill *will* pay dividends. In the separating department are huller-knives sharp, and the screens efficient? Does this mill send regular samples to a capable chemist to test separation efficiency or does the superintendent just guess it is all right? Weekly samples are not sufficient check, a sample from each shift-run should be analyzed promptly so that any inefficiency in separation may be detected quickly. We must concede that most mills follow this practice at present. Is the roll-grinding to uniform mesh? If not, the oil left in the cake will be high.

This brings us to the cookers. The proper cooking of seed is an art which can be standardized by the application of recording thermometers and temperature control apparatus to the cookers. Given these aids to operation, one man can handle much more work than formerly and still obtain efficient results in pressing.

Press-Room Practice

It is unthinkable to believe that any press-room is operated today without automatic cake-former, accumulator or automatic change-

valves, but a census of mills would probably reveal some without one or more of these essentials to economic operation. It behooves the progressive cottonseed miller to give serious consideration to the problem of press-loading and discharge, which are the operations accounting for most of the press-room labor.

If the crushers themselves create a demand for mechanical equipment to lighten the burden of this labor, our wide awake press-room machinery manufacturers will certainly provide such equipment.

Press-cloth expense can be kept at a minimum only by strict attention to careful forming of loads, uniformity of pressure and rigid inspection and repair of press-cloths and presses.

Cake-trimming must be well regulated, for it is just as expensive to trim too deep and thus rework stock unnecessarily as it is not to trim at all.

Now let's consider the best cake produced in the most modern mills. After exercising the greatest care possible throughout their mills as outlined above the best crushers in the country are selling cake or meal at thirty to forty dollars a ton which contains anywhere from five to seven percent of oil worth about one hundred and eighty dollars a ton.

When the oil is extracted from the cake by means of a volatile solvent the resulting nineteen hundred pounds of cake is worth just as much as the original ton because of increased protein value after extraction.

We are informed from the most reliable sources that the cost of operating an extraction plant including power, labor and fixed charges will not exceed $4.50 per ton on a twenty-five ton plant. Here is a profit of $4.50 a ton of cake for every oil miller, over $100 a day for a four press mill. European crushers take this profit and in the competitive struggle American crushers will be forced to eventually. WHY NOT NOW?

Quality of Oil

And now we come to consideration of the primary, most valuable product of the mill; the oil, the very reason for the mill's existence.

Our most advanced and progressive millers spend much time and effort and many dollars to perfect their seed handling and separating apparatus and methods in order to increase the yield and quality of oil, then, when they have succeeded in winning this, their primary product, from nature's storehouse, let it stand in the tanks in contact with meal, gummy and resinous matters which act as ferments, causing the quality to deteriorate steadily and rapidly.

Under the trading rules this practice costs the crusher many good dollars which he can easily put in his pocket by the installation of inexpensive equipment for the filtration of his oil. Since the passage of the new oil settlement rule this journal has repeatedly shown in its columns the profits which occur to the miller from filtration of his oil. Many progressive millers have installed oil-filtration equipment already this season, and it is a safe prophecy that all mills will eventually have such equipment. Again we must ask, WHY NOT NOW? In closing we cannot refrain from quoting the words of T. J. McNulty, of Brookhaven, Mississippi, who says: "One of the drawbacks of the oil mill industry is that it takes *too long* for us to take up and use methods that will save us money."

The American Oil Chemists' Society

Notes and Correspondence

Letter to Members from President Trevithick

Dear Fellow Members:

The Holiday season being with us again, permit me to extend most cordial Christmas Greetings and best wishes for a most prosperous and happy New Year to you all.

The Society year is fast drawing to a close, and it is desired that the various Committees finish their work as soon as possible. Their recommendations should be in the hands of the Uniform Methods Committee not later than April 1st, in order that they can be properly considered before the annual meeting. Will each of the Chairmen send one copy of his report to the Chairman of the Planning Committee, Mr. H. J. Morrison, Ivorydale, Ohio, one copy to the Chairman of the Uniform Methods Committee, Mr. N. C. Hamner, Dallas, Texas, and one copy to the President.

So far this year, the officers of the Society have been very busy trying to get some results in the standardization of Lovibond Glasses. Miss Walker became research associate at the Bureau of Standards in September, and besides assisting in standardizing the set of 130 red glasses, has been preparing a set of glasses which are accurate and are as nearly as possible actual units or tenths of units. These will make much easier the task of checking the actual values of the glasses to be submitted by the members. We hope to announce very soon that the Bureau of Standards will be ready to start on our glasses.

The Bureau has finished the work on the red glasses, and although Mr. Priest promised only to pick out the actual 7.6 glasses from those submitted, his staff have accurately calibrated the glasses and returned them to us with the correct values of each glass. This entailed a great deal more work by the Bureau, but it gave us 130 accurately standardized glasses, instead of just 20 or 25 glasses which were 7.6 and the rest of unknown values. As soon as the Bureau staff have prepared their set of glasses of actual tenth unit values, they will calibrate the yellow glasses submitted, by obtaining the correction of red for them. As Mr. Priest reported in Memphis, most of the yellow glasses are not true yellows but each contains red equivalent to one or more tenths.

The Revision Committee has about completed its work of revising the methods of the Society by eliminating all references to the Interstate rules, etc., in them and making them strictly chemical methods. By the time of the annual meeting if not before, we hope to have a complete report from this committee, and also to have some plan ready for publishing the methods complete, probably in a loose-leaf volume. This volume should contain not only the methods formerly published in the Interstate Rule Book, but also the "Fat Analysis Committee" methods and such other methods as oil chemists find necessary and useful in their work. By having the methods in loose leaf form, we can keep them up to date with the annual revisions.

In closing, I desire to extend the

President's thanks to all Chairmen and members of committees and to the membership at large for their splendid co-operation in the Society's work and to bespeak your continued efforts for the progress of our profession and of the oil industry.

H. P. Trevithick, Pres.,
December 7, 1927

Progress on Color Glass Standardization

The President of the American Oil Chemists' Society has received the following correspondence from the Bureau of Standards, Department of Commerce, reporting on the status of the Lovibond color-glass standardization work, as of October 1, and on the further progress attained in October. As outlined in the reports, it is the purpose of the Bureau to submit a monthly statement of progress to the Society.

Mr. H. P. Trevithick, President,
American Oil Chemists' Society,
New York Produce Exchange,
No. 2 Broadway,
New York, N. Y.

Subject: Standardization of Lovibond Glasses.

Dear Sir:

1. I am transmitting herewith report on status of cooperative work on standardization of Lovibond glasses as of October 1, 1927.

2. Another copy of the same report is being sent to Mr. Putland.

3. Your attention is particularly invited to Mr. Priest's recommendation that either yourself or Mr. Putland (or both of you) make regular monthly visits to the Bureau in order to keep in close touch with the progress of this investigation. I hope you will find it possible to follow this suggestion.

Respectfully,
L. J. Briggs,
Acting Director
Bureau of Standards
Department of Commerce.

Report on Status of Work, October 1, 1927

1. Miss Geraldine K. Walker, employed as Research Associate by the American Oil Chemists' Society, reported for duty at the Bureau of Standards Monday morning, September 12, 1927.

2. Status of Standardization at Time of Miss Walker's Arrival:

(1) The specifications for American Standard Red and Yellow Scales had been established by Priest and Gibson. (Reported at Memphis Convention, A.O.C.S., May, 1927.)

(2) The following glasses had been standardized in terms of this scale:

(a) In Bureau of Standards set (B.S. 9940) glasses of *nominal* values as follows: Red glasses of integral values from 1.0 to 20.0; yellow glasses of integral values from 1.0 to 20.0: fractional red glasses from 0.01 to 0.09, and the exact tenths from 0.10 to 0.90; fractional yellow glasses, 0.01, 0.10, 0.20, and 0.40.

(b) Glasses belonging to A.O.C.S. (B. S. Test No. 41960): Red glasses, nominally integral values from 1.0 to 20.0, and nominally exact tenths from 0.10 to 0.90; yellow glasses, nominally integral values from 1.0 to 10.0, and 20.0, 30.0, and 35.0.

(Results on the A.O.C.S. glasses were reported at Memphis Convention, A.O.C.S., May, 1927.)

(3) During July, Priest, with Judd's assistance, had made a thorough study of the relations between the transmission of the yellow glasses for a certain few particular wavelengths and the sunlight transmissions and correct Lovibond numerals of the same glasses. These relations will be useful in routine calibration of the yellow glasses.

(4) During July, Priest also made a preliminary study of the project of standardizing the red glasses alone without reference to the 35-yellow.

(5) The following glasses submitted by the A.O.C.S. (through Mr. Trevithick) were at the Bureau September 12:
66 35-yellows submitted August 4, 1927; 124 reds submitted August 4, 1927; 1 35-yellow submitted by letter of August 27, 1927; 3 7.6 reds submitted by letter of August 27, 1927.

(6) Preliminary determination of the red corrections to be applied to each of 64 of the above mentioned 35-yellow glasses had been made by Judd in August. These corrections have not yet been verified.

(7) A preliminary sorting of 123 of the above mentioned red glasses, for the purpose of selecting a group between true values 7.5 and 7.7 had been carried out by Priest, Judd and Riley. In this sorting, errors as large as O.7R in the nominal values as submitted have been discovered.

(8) Work on selecting red glasses of exactly integral values at unit intervals and exact tenths at intervals of one-tenth had been started. This selecting was undertaken in order to provide a *convenient* set of "working standards" for routine calibration of other glasses.

3. Progress by Miss Walker, September 12-30:

(1) The equivalents of 129 red glasses (submitted by Mr. Trevithick, August 4, August 27, and September 24) have been determined in terms of the scale of Priest and Gibson. It is highly probable that nearly all of these equivalents are correct to within about 0.1R but they ought to be verified before being finally reported. Errors as large as 0.7R in the nominal values as submitted have been found. (The calibration of these 129 glasses occupied about 57 actual working hours.)

(2) About 1/6 of the task of selecting red glasses for exact integers and exact tenths has been completed.

(3) Determinations of the spectral transmissions of the following glasses from the B. S. Set 9940 have been made: 1.0, 0.84, 2.8, 3.8.

4. Cooperation of other Members of Staff, September 12-30:

(1) Gibson, Judd, and Riley have continued work on the selection of red glasses for exact integers and tenths.

(2) Priest and Judd have prepared the preliminary draft of report on "Selection of a Group of Correct Lovibond 7.6 Red Glasses from a collection of Red Glasses of Nominal Values between 7 and 8." (This will have to be revised.)

5. In Regard to Future Reports:

It is expected that a monthly report of progress will be prepared at the end of each month to be forwarded to Mr. Trevithick and Mr. Putland.

However, it will be very difficult to make all matters clear by such written reports. In order that the Society may be more fully advised than is possible by such

written reports, I strongly recommend that either Mr. Trevithick or Mr. Putland, or both make regular monthly visits to the Bureau beginning about December 1st. (I will probably make an oral report at the New York Meeting of the Society, October 28th). The first Saturday in each month would be a date agreeable to me for such visits.

Report for October, 1927

1. The preliminary selection of individual red glasses to represent as accurately as possible exact tenths from 0.1 to 0.9 inclusive and exact integers from 1 to 11 has been completed by Gibson, Judd, Walker and Riley. Unfortunately, there were some cases in which no one glass could be found in the Bureau set (B.S. 9940) to represent the exact integral values. These cases will require further attention. Exact integral values can probably be realized by combining two glasses.

2. In the program of verifying by spectral transmission, the values of glasses selected by color match, the spectral transmissions of eleven more red glasses (0.84, 2.8, 3.8, 5.4, 6.8, 7.8, 8.8, 9.8, 0.11, 0.17, 0.18, maker's numerals from B.S. 9940) have been determined by Walker and Gibson.

3. The values of eight more glasses (0.84, 2.8, 3.8, 5.4, 6.8, 7.8, 8.8, 9.8, maker's numerals from B.S. 9940) have been computed by Riley, under Gibson's supervision, on the new scale of Priest and Gibson.

4. Further tests of the additivity of values of red glasses on the new scale of Priest and Gibson have been made by Judd and Walker by the method of direct comparison of colors in the Martens photometer. All of these tests indicate that the errors in the values assigned to individual glasses by Priest and Gibson are less than 0.1. The method of direct comparison of colors is not adequate to detect errors smaller than this.

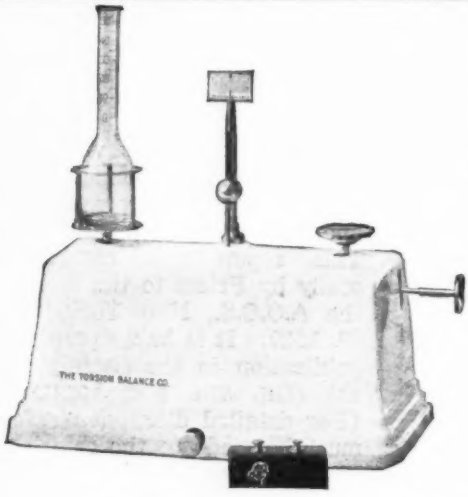
5. Report on the calibration of 129 red glasses submitted by the A.O.C.S. has been prepared by Judd and Walker as planned by Priest. This report was communicated orally by Priest to the meeting of the A.O.C.S., New York, October 28, 1927. It is being prepared for publication in the Society's Journal (OIL AND FAT INDUSTRIES). (For detailed discussion reference must be made to the complete report.) These glasses have been returned to Mr. Trevithick.

6. At the meeting of the A.O.C.S., New York, October 28th, Priest reported on the tests of color sense of oil chemists made at the Memphis convention, May, 1927. This report dealt particularly with the sensibility to differences in Lovibond red at 35-yellow 7.6 red. It is being prepared for publication.

7. Priest has planned a new method of using the Arons chromoscope to grade the red glasses. Preliminary tests of it have been made. It is expected that this method will afford an independent check on the calibration by spectral transmission and may perhaps be developed into a rapid routine method for regarding the red glasses submitted for test.

Irwin G. Priest,
Chief Colorimetry Section.

It is with regret that we announce the death, on October 29, at Fresno, California, of Robert Hulme, a member of the American Oil Chemists' Society and a well known oil chemist.



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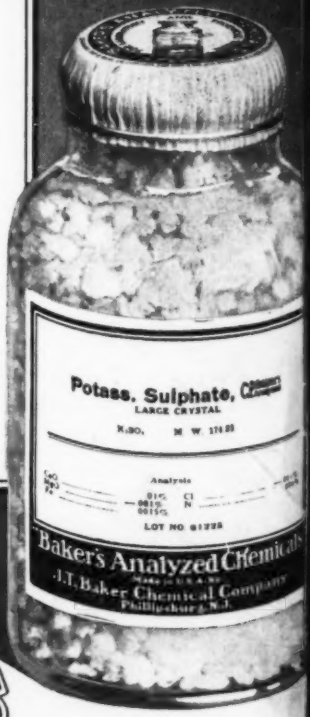

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